Synthesis, characterization and formulation of Diammonium ethylene bis(5-nitroiminotetrazolate)

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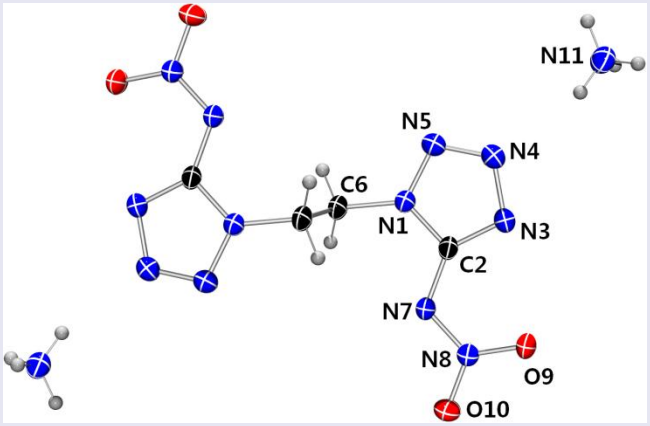
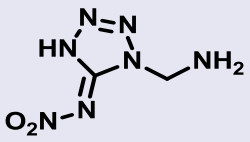
Overview

- ❖ High Nitrogen explosives
- ❖ Tetrazole derivatives
- ❖ Nitroiminotetrazole
- ❖ Enhanced Synthesis method
- ❖ Characterization of EBNIT
- ❖ Synthesis of Salt-EBNIT
- ❖ Characterization of Salt-EBNIT
- ❖ Summary



High Nitrogen explosives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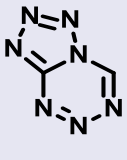
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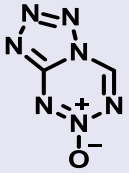
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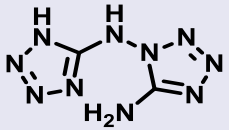
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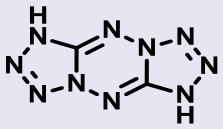
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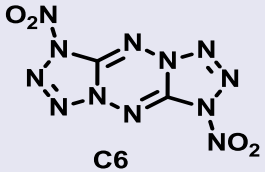
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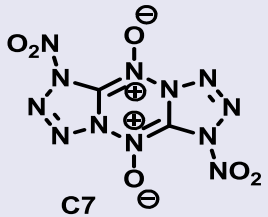
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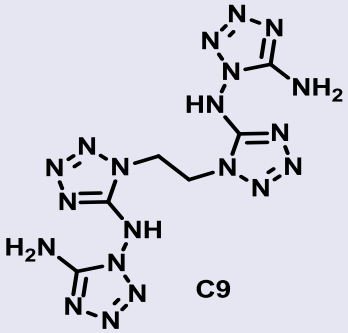
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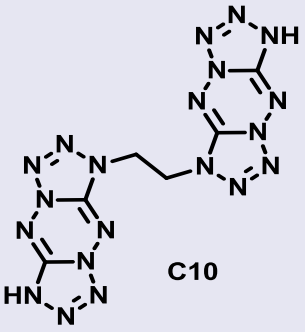
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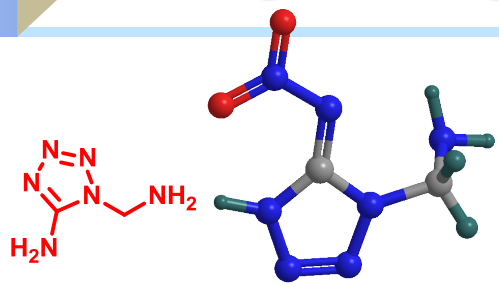
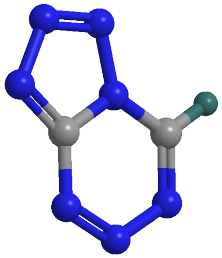
High Nitrogen explosives, Tetrazole derivatives



C1

ΔH_f^o= 46.1 kcal mol^{-1}
d = 1.635 g cm^{-1} (1.960)
Impact Sensitivity = 16.2 J
P = 22.73 GPa (27.77)
D = 7948 m s-1 (8487)
Oxygen Balance = -45.3%
N Content = 61.6%



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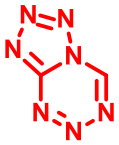
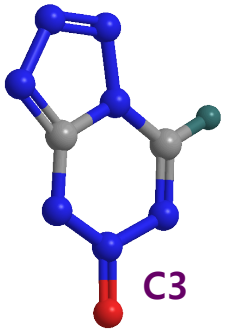


ΔH_f^o= 151.6 kcal mol^{-1}
d = 1.859 g cm^{-1} (1.847)
Impact Sensitivity = 5.4 J
P = 38.47 GPa (37.75)
D = 9323 m s-1 (9277)
Oxygen Balance = -58.5%
N Content = 79.7%



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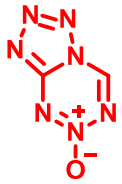


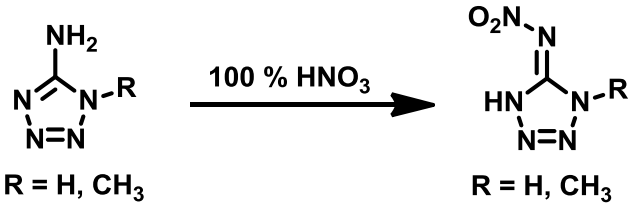
ΔH_f^o= 168.9 kcal mol^{-1}
d = 1.918 g cm^{-1} (1.905)
Impact Sensitivity = 3.5 J
P = 40.82 GPa (40.07)
D = 9306 m s-1 (9262)
Oxygen Balance = -40.3%
N Content = 70.5%

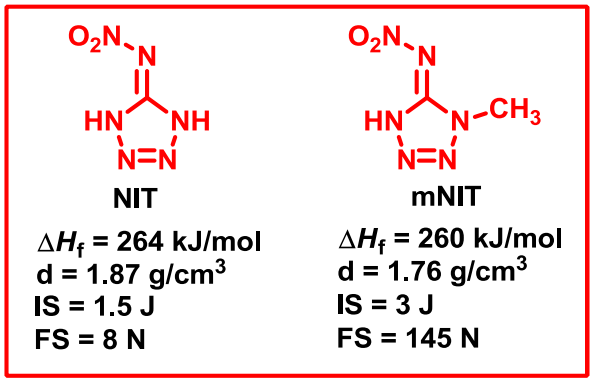
TNT
ΔH_f^o= -15.1 kcal mol^{-1}
d = 1.654 g cm^{-1}
Impact Sensitivity = 40.0 J
P = 21.00 GPa
D = 6930 m s-1
Oxygen Balance = -74.0%
N Content = 18.5%

RDX
ΔH_f^o= 16.5 kcal mol^{-1}
d = 1.806 g cm^{-1}
Impact Sensitivity = 6.5 J
P = 33.80 GPa
D = 8700 m s-1
Oxygen Balance = -21.6%
N Content = 37.8%

HMX
ΔH_f^o= 17.9 kcal mol^{-1}
d = 1.905 g cm^{-1}
Impact Sensitivity = 7.3 J
P = 39.40 GPa
D = 9110 m s-1
Oxygen Balance = -21.6%
N Content = 37.8%

Nitroiminotetrazole



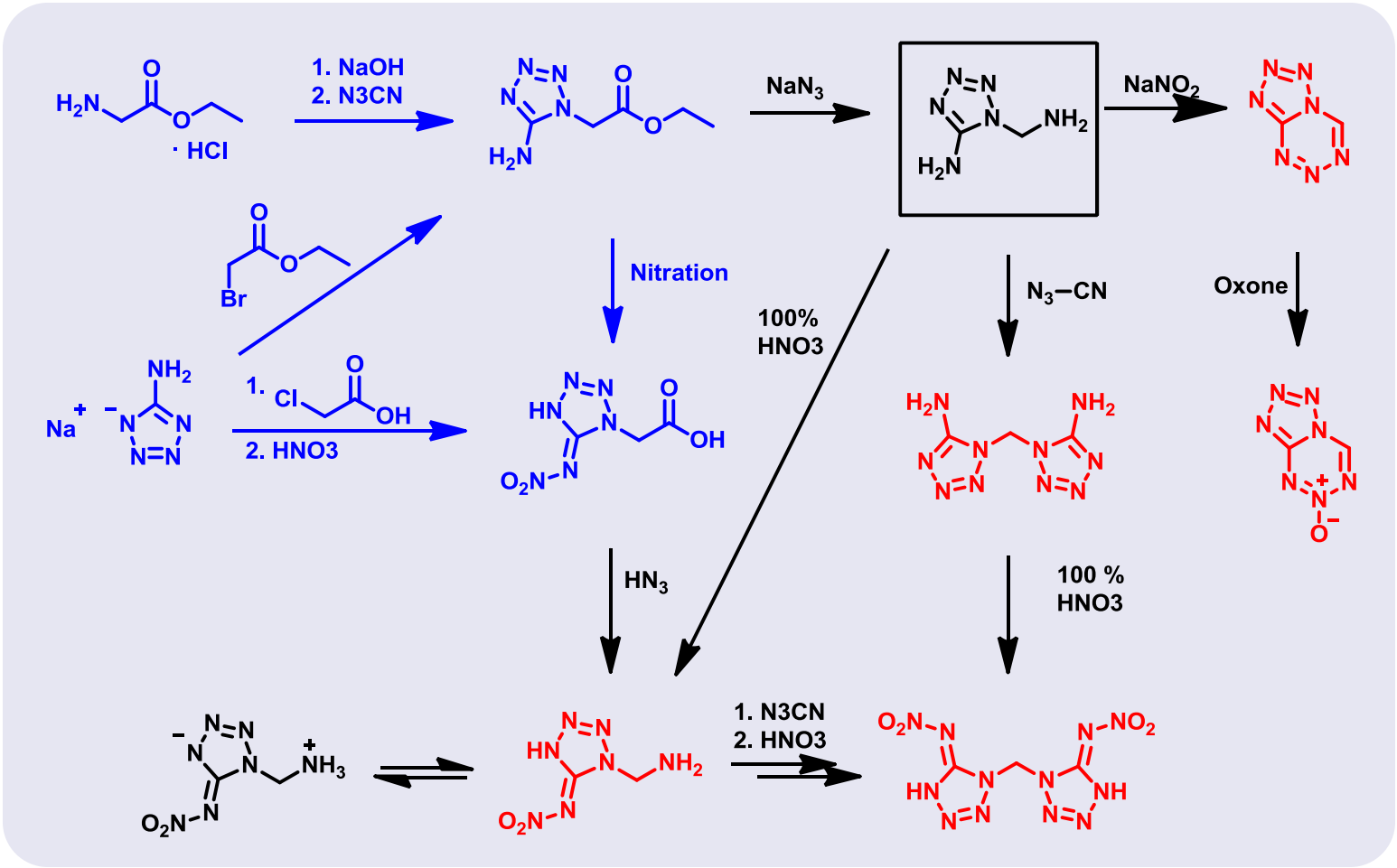


Explosives	Density [g cm^{-3}]	*P* [GPa]	*D* [m s^{-1}]	IS [J]
TNT	1.65	19.5	6881	15
RDX	1.82	35.2	8977	7.4
NIT	1.87	36.3	9173	1.5
mNIT	1.76	29.5	8433	3

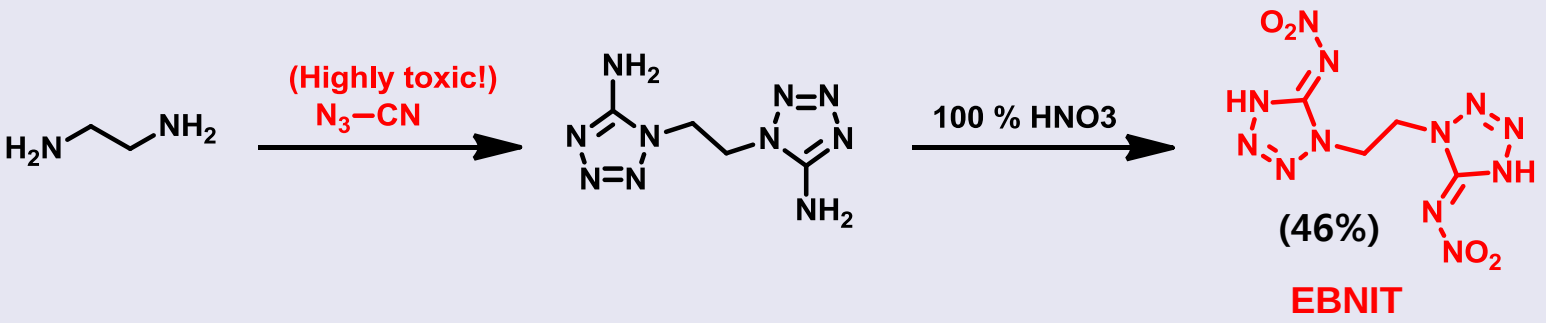
metal complex
(primary explosives)
or
energetic salts
(secondary explosives)

Klapötke, T. M.; Stierstorfer, J. *Helv. Chim. Acta* **2007**, *90*, 2132-2150.

Bridged Tetrazole derivatives synthesis

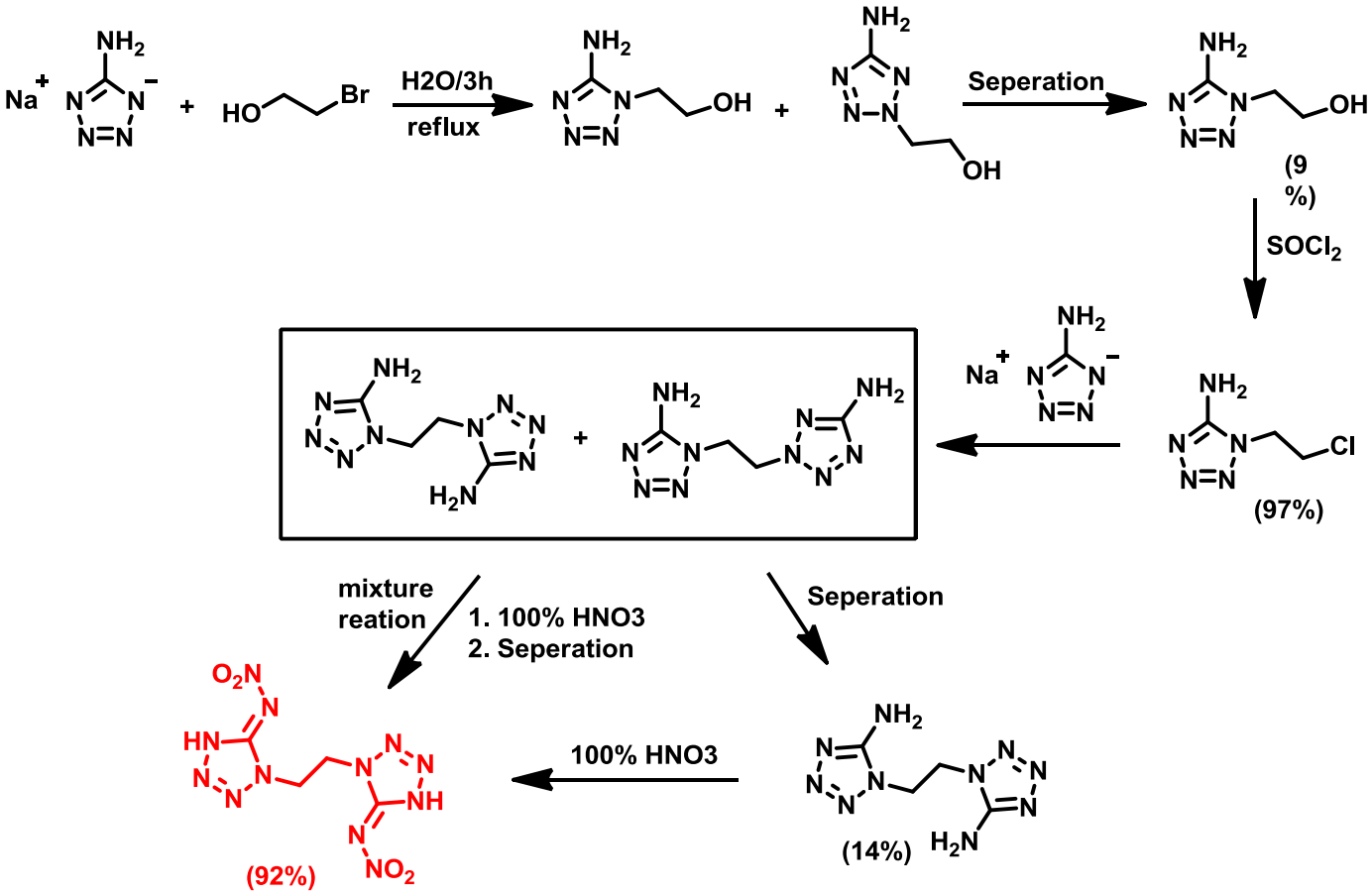


Old Synthesis method (Bistetrazole)

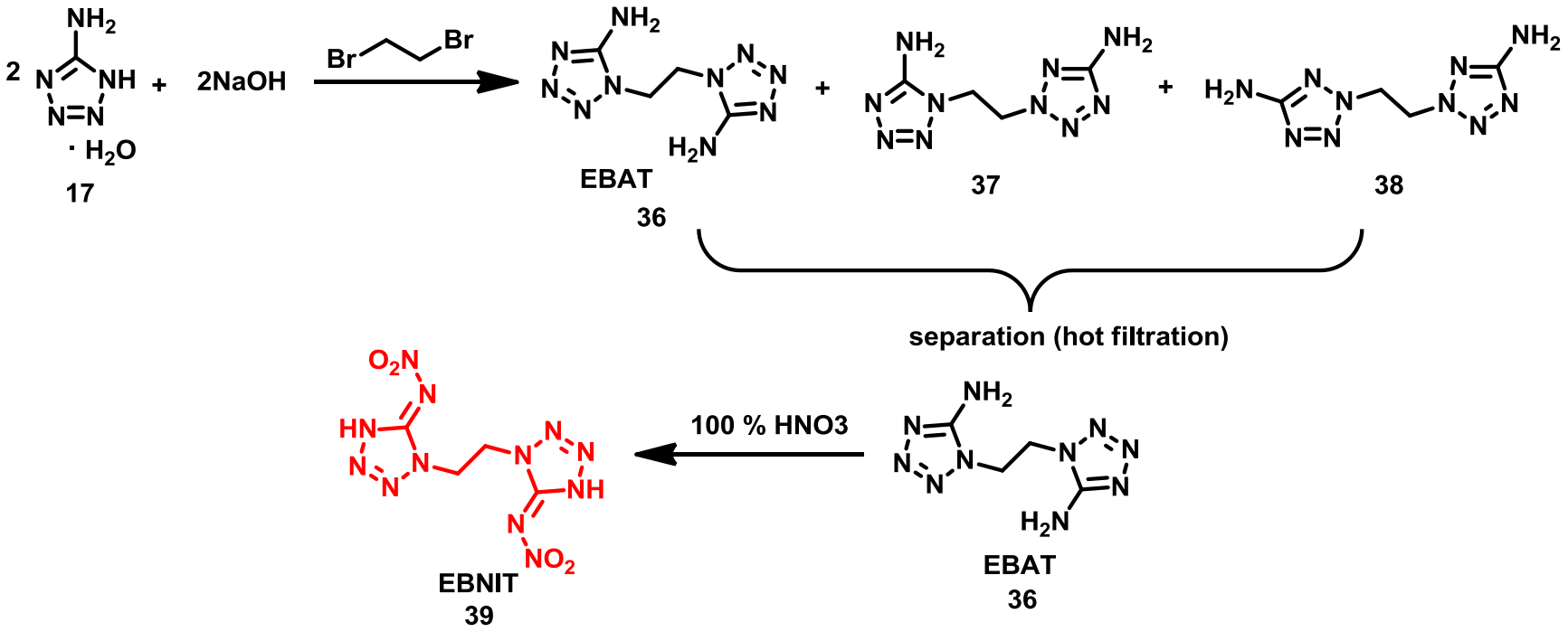


Joo, Y. –H.; Shreeve, *J. M. Angew. Chem. Int Ed.* 2009, 48, 564

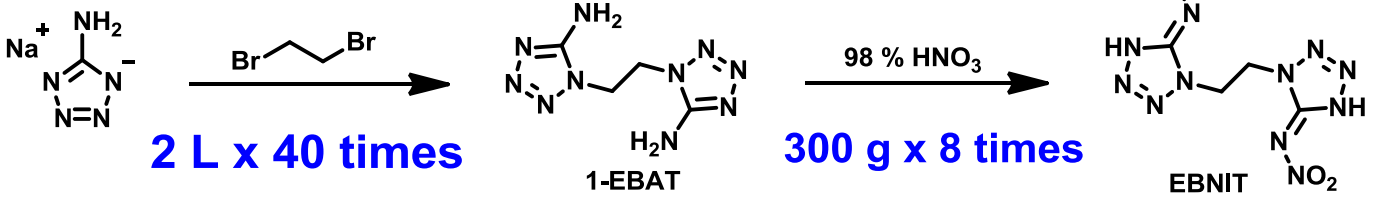
Synthesis method of small scale in Lab.

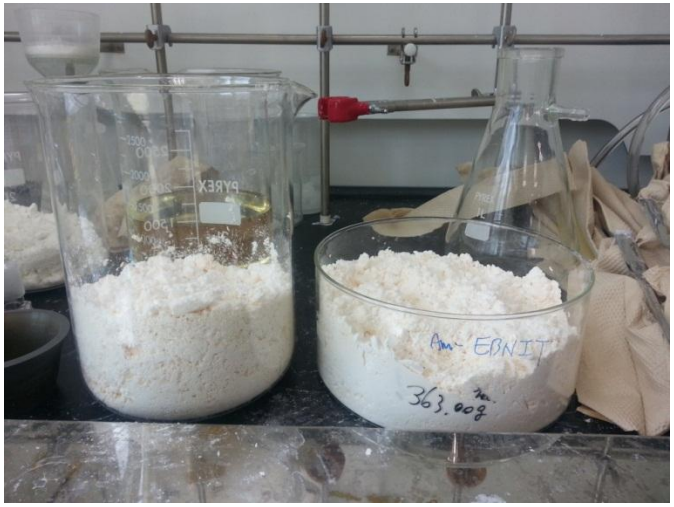


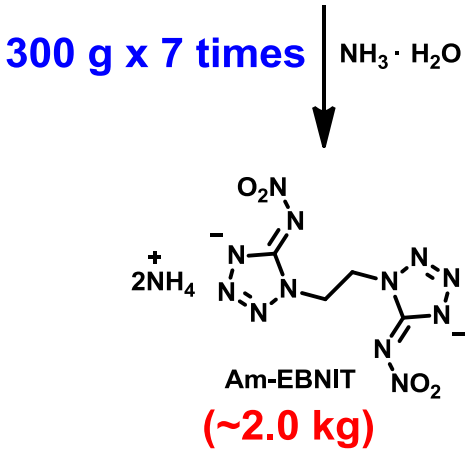
Synthesis method of scale up in Lab.



Scale up Synthesis in Lab.

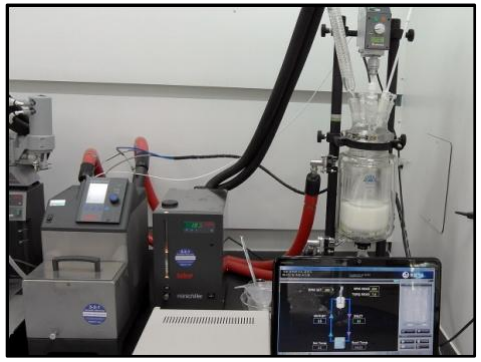






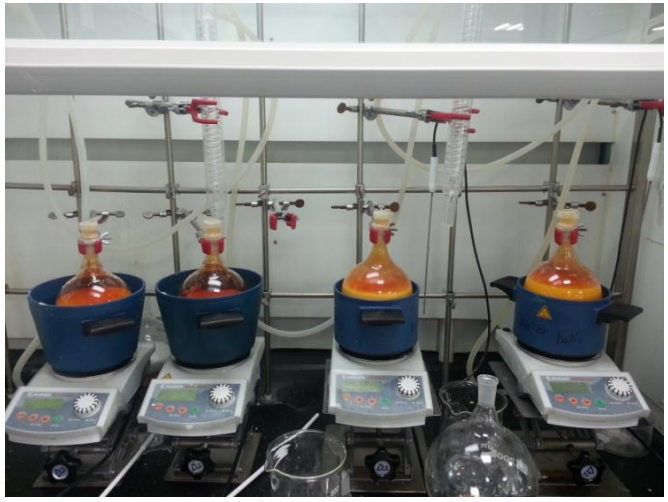
Scale up Synthesis in Lab.

EBAT 50g synthesis



EBAT scale up research

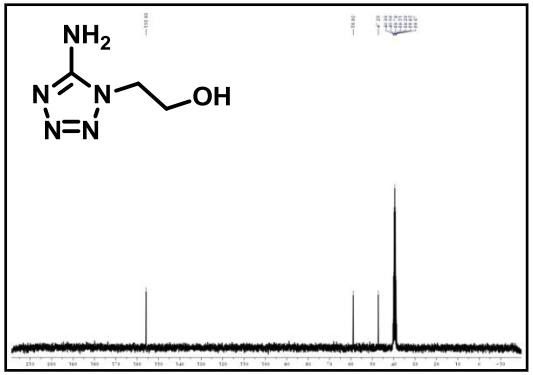
EBAT hot filtration process

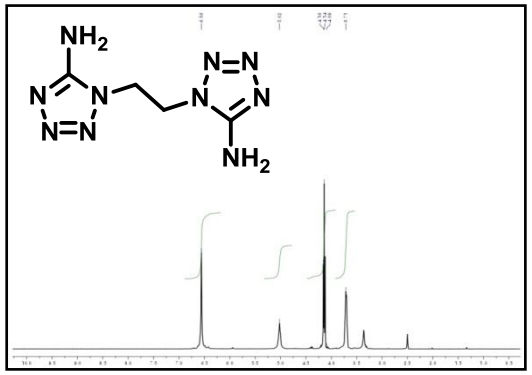


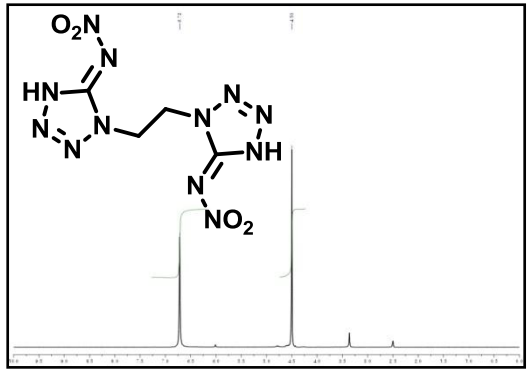
EBNIT nitration synthesis

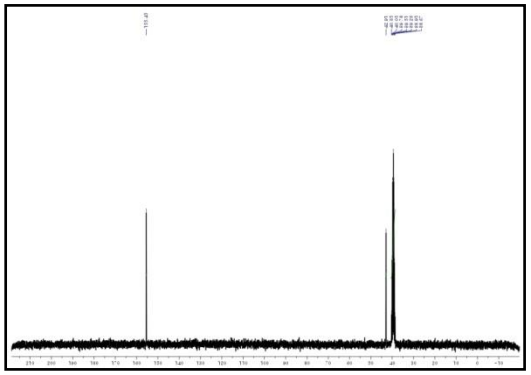
Am-EBNIT synthesis

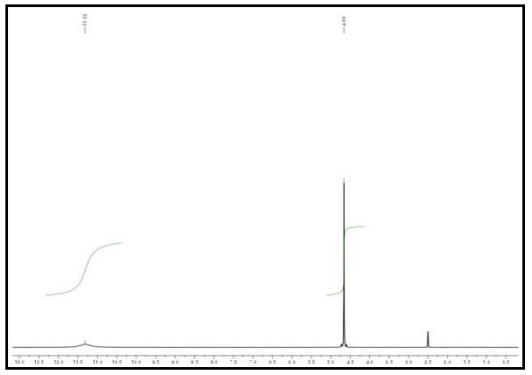
NMR of Synthesis materials

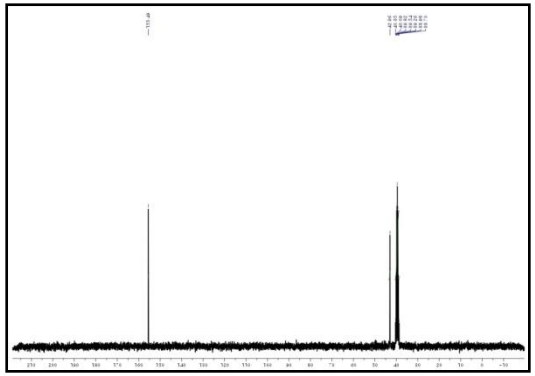




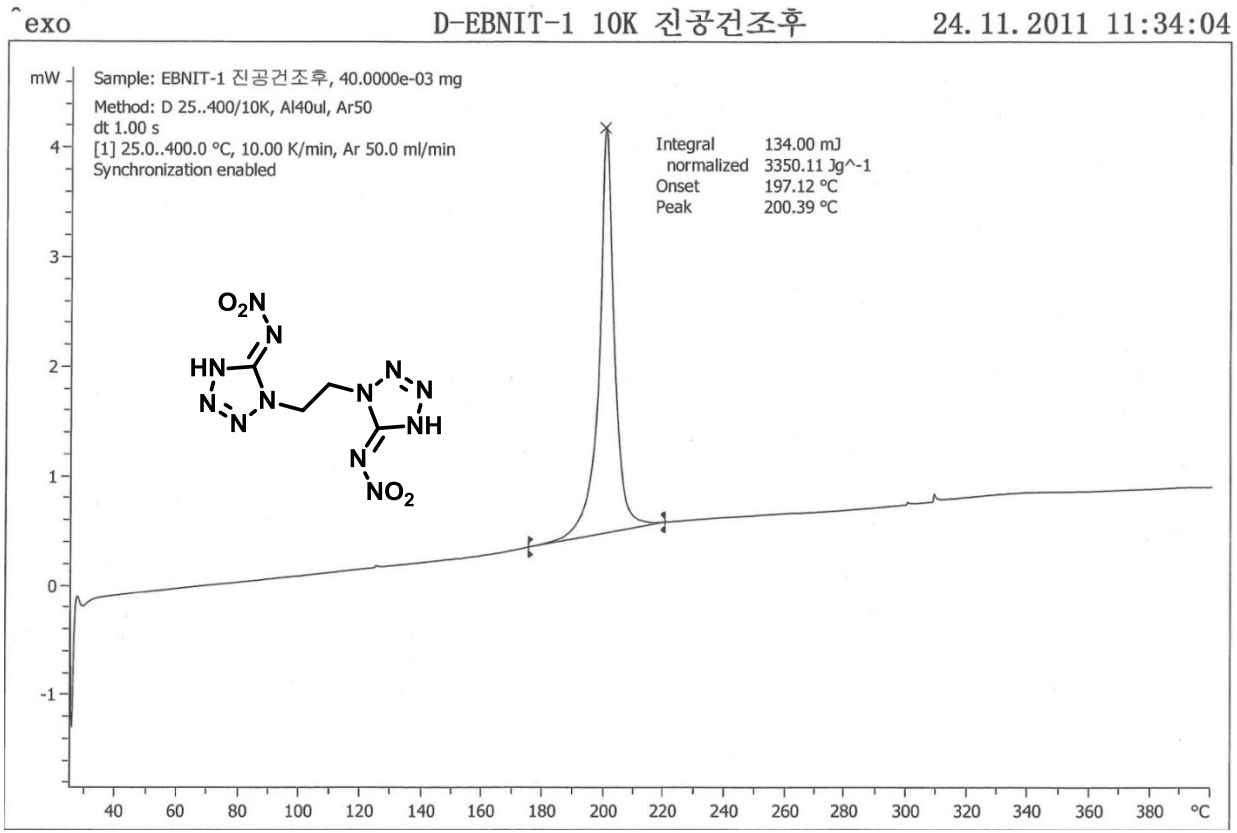






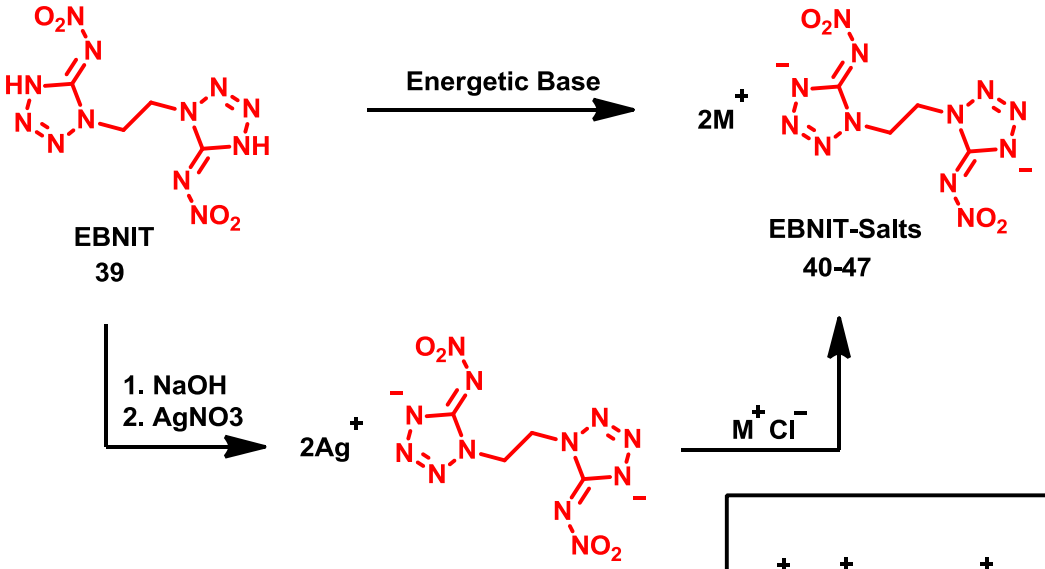


DSC of Synthesis materials



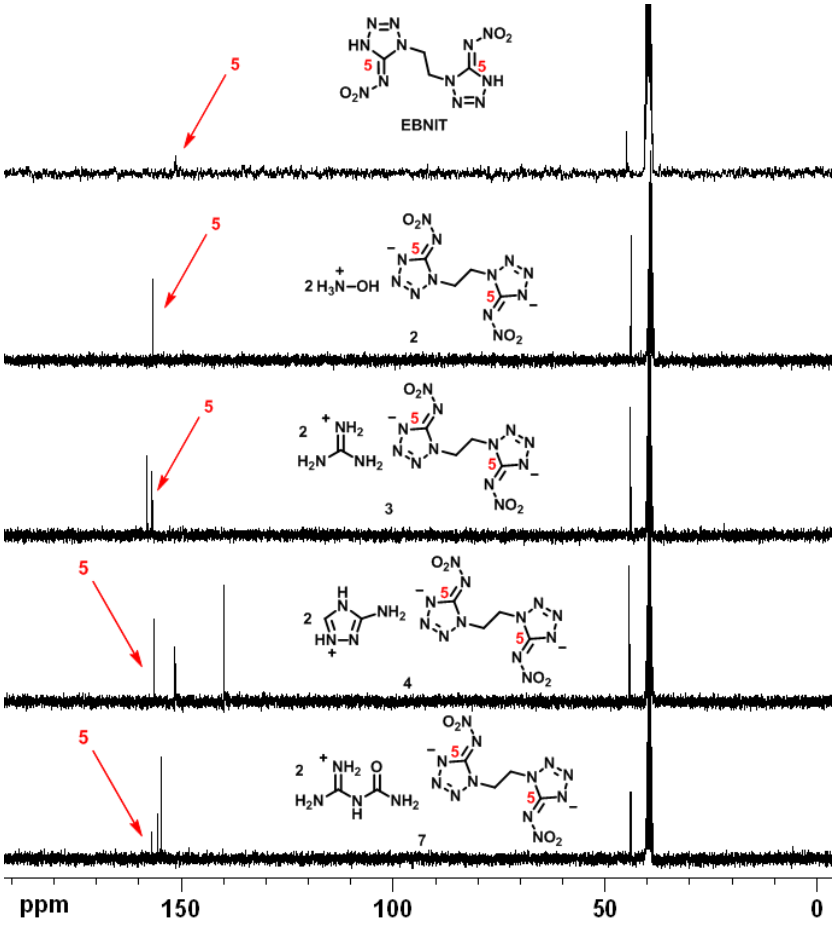
DSC curve of 1,2-bis(5-nitroiminotetrazol-1-yl)ethane

Salts synthesis of EBNIT

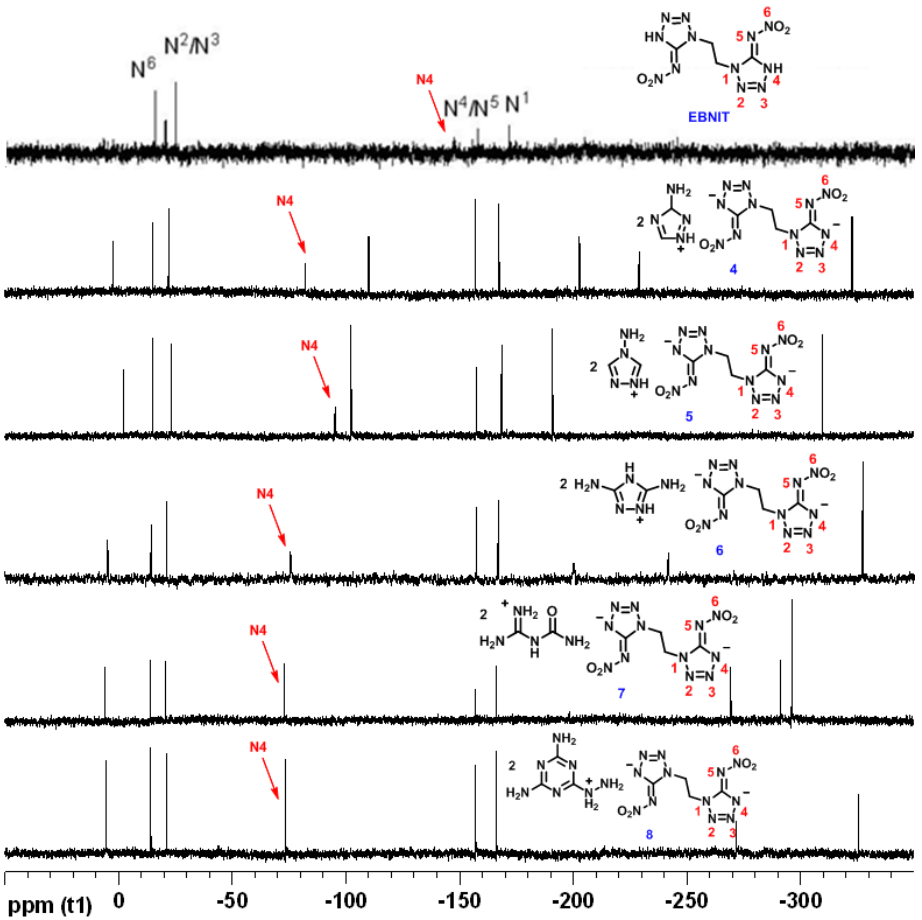


NMR analysis of EBNIT salts

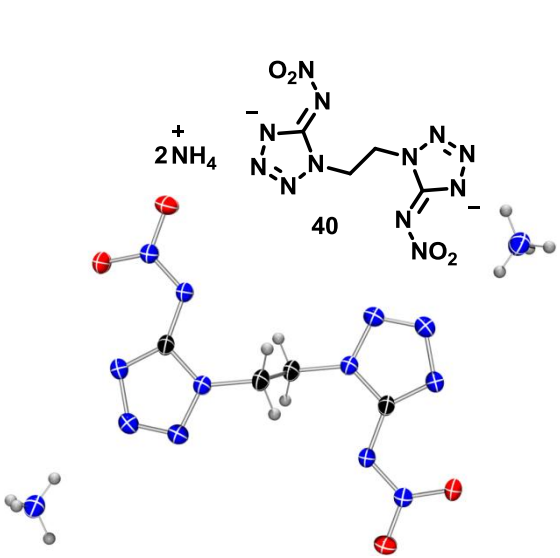
^{13}C-NMR

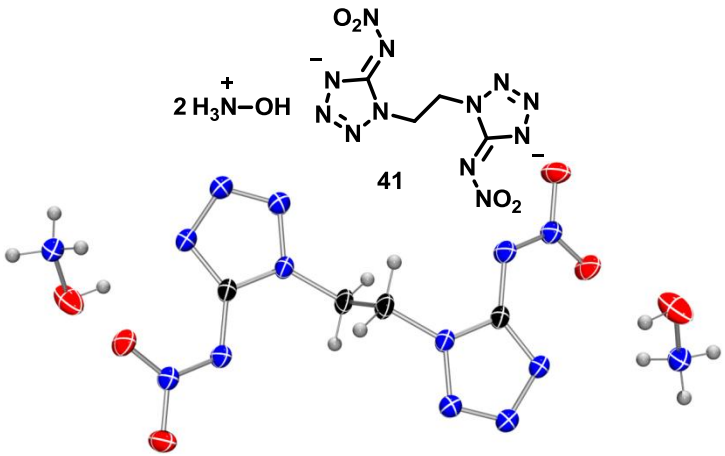


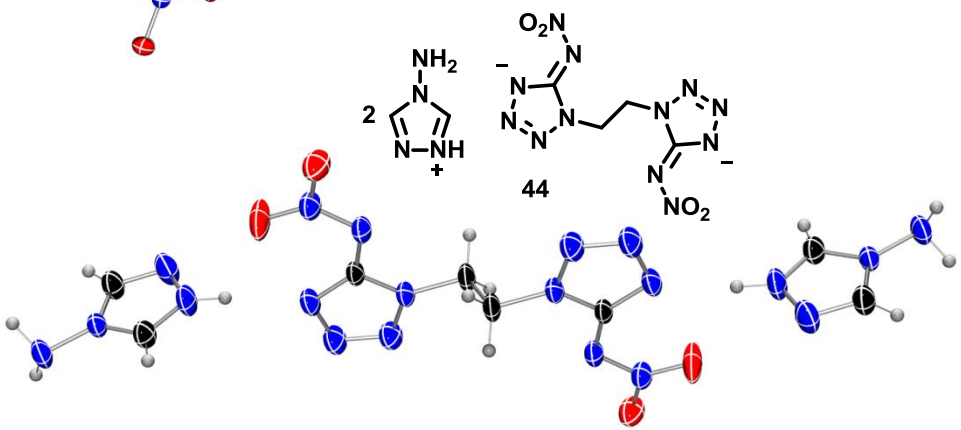
^{15}N-NMR



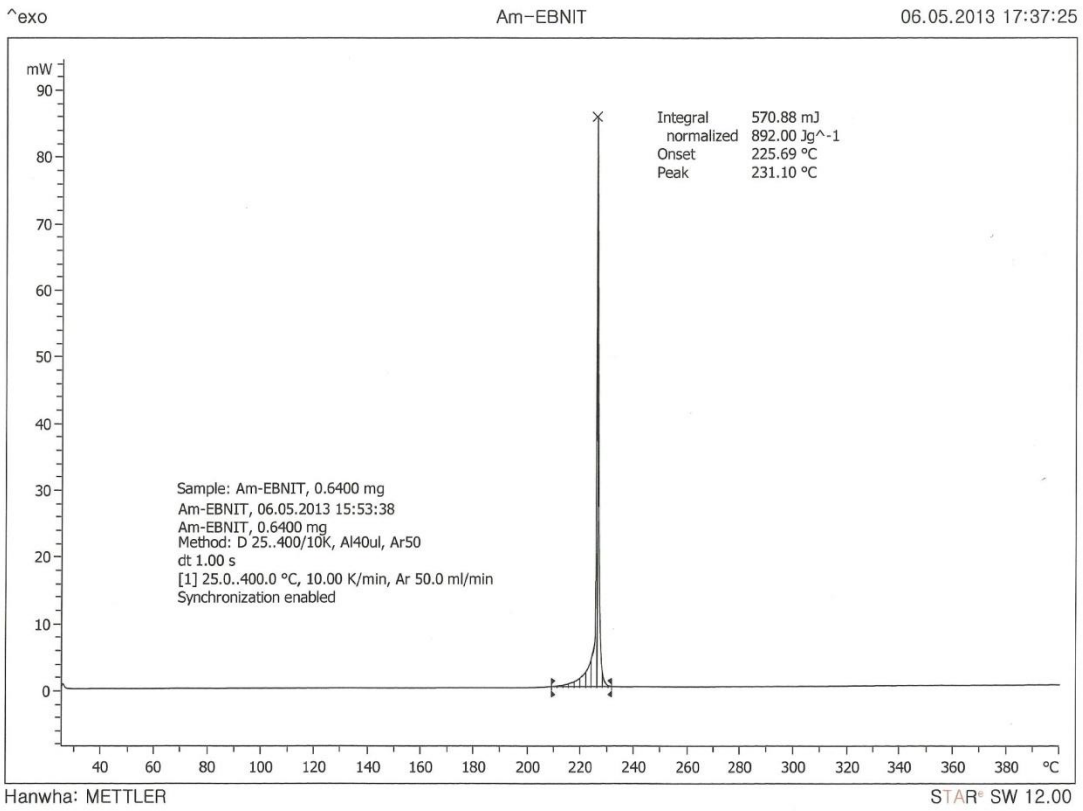
Crystal structure of EBNIT salts



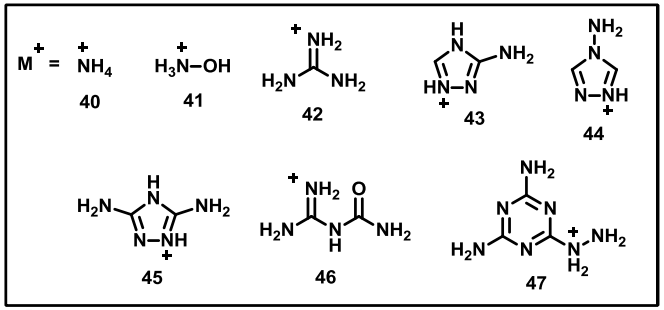


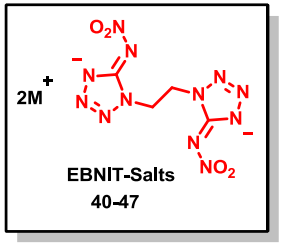


DSC of Synthesis materials Am-EBNIT



Properties of EBNIT salts

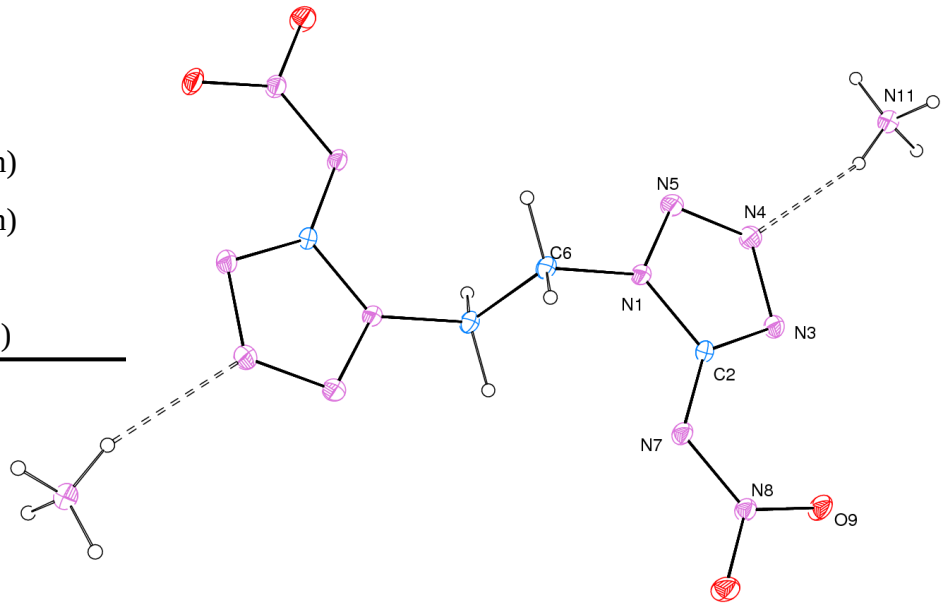




Compound	Nitrogen contents (%)	Decomposition [°C]	Crystal Density [g cm^{-3}]	Heat of formation [kJ mol^{-1}]	C-J pressure [GPa]	Detonation velocity [m s^{-1}]	Impact Sensity [J]	Friction Sensity [N]
40	61.2	202	1.754	730	33.26	8979	4	180
41	55.7	210	1.807	849	38.16	9368	4	108
42	62.0	260	1.635	772	25.81	8213	40	360
43	61.4	223	1.676	1268	27.26	8311	10	120
44	61.4	204	1.661	1536	28.36	8455	6	168
45	63.4	244	1.747	1197	29.36	8537	6	216
46	56.9	240	1.857	890	33.50	8934	>40	>360
47	63.8	259	1.833	1362	31.34	8701	>40	>360

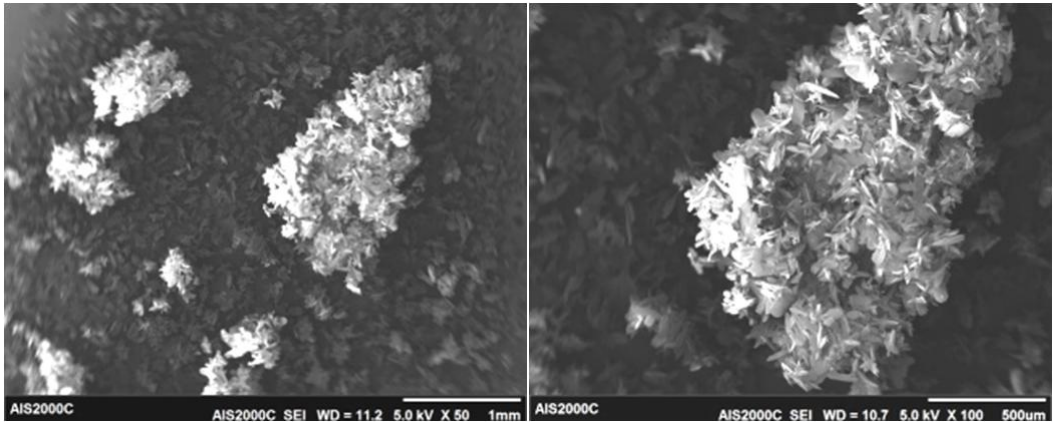
Properties of Am-EBNIT

Nitrogen contents(%)	66.2
Heat of formation(kj/mol)	730
Melting Point(**°C**)	202(decomposition)
Crystal Density (g/cm^{-3})	1.754
Detonation velocity(m/sec)	8,979 (Explo-5 estimation)
Detonation pressure(GPa)	33.26 (Explo-5 estimation)
Impact Sensitiveness(J)	4.83 (RDX CL-1 7.38)
Friction Sensitiveness(N)	268.5 (RDX CL-1 142.92)

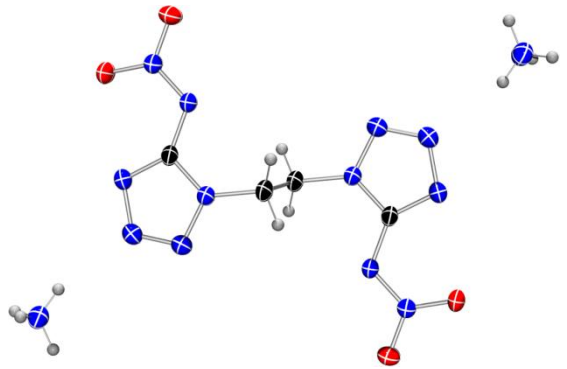


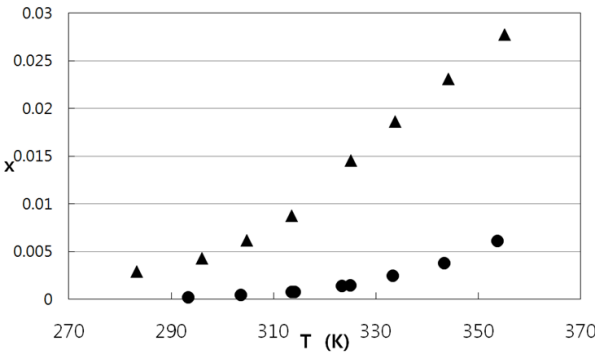
Single crystal x-ray diffraction analysis

Recrystallization of Am-EBNIT

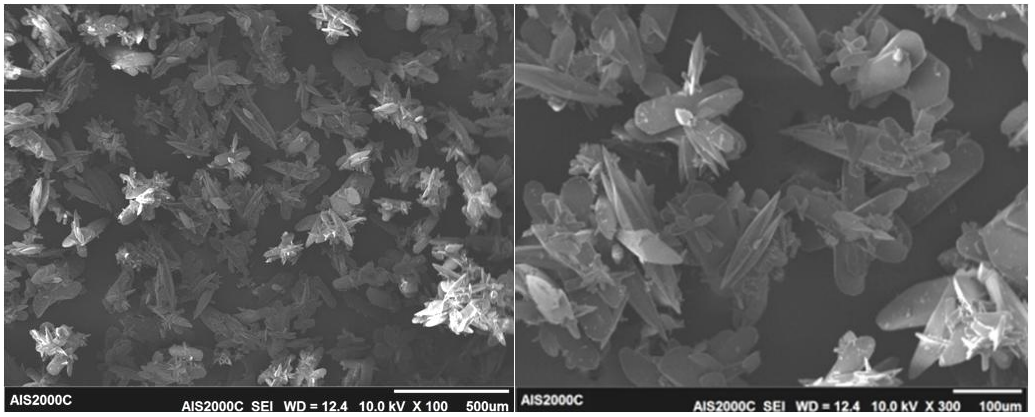


SEM of crude Am-EBNIT





Solubility in water of Am-EBNIT (▲)



SEM of recrystallized Am-EBNIT

Am-EBNIT Formulation

- **Prepare coated Am-EBNIT for pellet pressing and performance/sensitivity evaluations.**
- **Am-EBNIT was coated with 4% Hytemp(1%DOA) through a solvent slurry coating process. (Sigma mixer granulation)**
- **1kg batch size.**
- **Am-EBNIT will be pressed.**
- **No optimization performed at this time. (Shape, particle size)**

Sensitivity	Bear	Coated
Impact Sensitiveness(J)	4.83	6.14
Friction Sensitiveness(N)	268.5	>352.8

Summary

- ❖ Am-EBNIT has been High Nitrogen explosives
- ❖ Enhanced Synthesis method
- ❖ Further development needed to optimize particle shape and size
- ❖ Evaluate performance(VoD, pressure) and sensitivity (SSGT)
- ❖ Evaluate Am-EBNIT for green energetic explosives